

<p align="center">LLNL Environmental Restoration Division Standard Operating Procedure</p>	<p align="center">TITLE: Sample Containers and Preservation</p>
<p>APPROVAL _____ Date _____</p> <p>Environmental Chemistry and Biology Group Leader</p>	<p>PREPARER: V. Dibley</p> <p>REVIEWERS: R. Brown*, T. Carlsen, E. Christofferson*, J. Duarte, B. Failor*, R. Goodrich, B. Hoppes*, G. Howard, S. Kawaguchi, G. Kumamoto, J. Martin**, and B. Ward*</p>
<p>APPROVAL _____ Date _____</p> <p>Division Leader</p> <p>CONCURRENCE _____ Date _____</p> <p>QA Implementation Coordinator</p>	<p>PROCEDURE NUMBER: ERD SOP-4.3</p> <p>REVISION: 2</p> <p>EFFECTIVE DATE: December 1, 1995</p> <p align="center">Page 1 of 13</p>

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1.0 PURPOSE

This procedure defines the sample volume, container, holding time, and preservation method requirements for water, waste, soil, sediment, and sludge sampling recommended by the Contract Analytical Laboratories (CALs) and the available regulatory guidance.

2.0 APPLICABILITY

This procedure is to be used as a general guide in choosing a sample container and method of preservation for environmental samples collected by the Environmental Restoration Division (ERD).

3.0 REFERENCES

- 3.1 U.S. EPA (1993), *Test Methods for Evaluating Solid Waste Physical/Chemical Methods*, SW-846, 2nd edition, Washington, D.C.

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- 3.2 U.S. EPA (1992), *40 Code of Federal Regulations*, Chapter 1, § 136.3, Table II, Washington, D.C.
- 3.3 U.S. EPA (1983), *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, Washington, D.C.

4.0 DEFINITIONS

Not applicable.

5.0 RESPONSIBILITIES

5.1 Division Leader

The Division Leader's responsibility is to ensure that all activities performed by ERD at the Livermore Site and Site 300 are performed safely and comply with all pertinent regulations and procedures, and provide the necessary equipment and resources to accomplish the tasks described in this procedure.

5.2 Sample Coordinator (SC)

The SC's responsibility is to supply the sampling personnel with requested analyses, sample containers, and type of preservation.

5.3 Sampling Personnel

Sampling personnel are responsible for handling the samples collected for ERD as specified in the following procedure.

5.4 Task Leader

When conducting sampling other than routine ground water, the Task Leader is responsible for supplying all necessary sampling information to the sampling personnel. For drilling, a work plan is issued that contains a sampling plan.

6.0 PROCEDURE

6.1 Discussion

- 6.1.1 Preservation methods are generally limited to pH control, chemical addition, refrigeration, and freezing. Methods of preservation are intended to: retard biological action, hydrolysis of chemical compounds and complexes, and to reduce the volatility of analytes and adsorption effects.
- 6.1.2 Plastic containers or lids and aluminum foil should NOT be used due to possible sample contamination from the phthalate esters and other hydrocarbons within the plastic and on the foil surface.
- 6.1.3 VOC vials should be EPA level 1 and have supporting certification documentation on file with the SC.
- 6.1.4 Sample containers should be filled so that the sample does not come into contact with the sampler's gloves, thus potentially causing contamination.

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- 6.1.5 In the determination of trace metals, containers can introduce positive or negative errors by contributing contaminants through leaching or surface desorption, and depleting concentrations through adsorption. Containers such as Borosilicate glass, linear polyethylene, polypropylene, or Teflon are used for collecting metals for all environmental media. If necessary, brass or stainless steel tubes can be used during drilling operations.
- 6.1.6 Attachments A and B list the appropriate containers preservation and volume of sample needed for frequently requested analyses. If an analysis is not listed, contact a QC Chemist for more information.
- 6.1.7 VOC vials should be EPA level 1 and have supporting certification documentation on file with the SC.

6.2 Office Preparation

- 6.2.1 The SC will supply a sampling schedule, or the Task Leader will provide a sampling plan which lists the area and well or location to be sampled, the requested analyses, the CAL the sample is to be sent, and other sampling information.
- 6.2.2 See Attachment A for aqueous matrices and Attachment B for solids/sediments to determine the container, preservation, and volume necessary for the requested analyses. These volumes and weights listed in Attachments A and B may vary between CALs, depending on their policies and procedures. Each CAL should be consulted prior to use.
- 6.2.3 The type of sample analysis collected determines the type of bottle, preservative, holding time, and filtering requirement. Calculate the number of each type of container required and gather sampling materials as listed in the appropriate sampling or drilling SOP.
- 6.2.4 Samples shipped via common carrier or U.S. Postal Service, must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR part 172), and is the responsibility of the person who prepared the material for shipment to ensure this compliance is met. For the preservation requirements of samples, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation (DOT) has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCL) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); nitric acid (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and sodium hydroxide (NaOH) in water solutions at concentrations of 0.08% by weight or less (pH about 12.3 or less). See SOP 4.4, "Guide to the Handling, Packaging, and Shipping of Samples."
- 6.2.5 The containers or preservation designated in the Attachments A and B are mandated by a specific analyses. Variations must be approved by the Environmental Chemistry and Biology Group Leader.

6.3 Field Preparation

- 6.3.1 The SC may acquire the pre-cleaned and preserved glass and polyethylene containers from the CAL.

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- 6.3.2 Obtain the appropriate containers from storage (Building 833 at Site 300), or the SC, CAL, or LLNL Stores. Place containers in the sampling cooler with packing material (to prevent breakage), ice (either loose, bagged, or Blue), and include a trip blank if VOCs are to be sampled. Blue ice should be bagged and used when shipping samples via Federal Express (see SOP 4.4, "Guide to the Handling, Packaging, and Shipping of Samples").
- 6.3.3 Trip blanks can be obtained from the refrigerator designated for trip blanks in Building 833 at Site 300, or from the Livermore Ground Water Monitoring SC.
- 6.3.4 The SC should keep a sufficient supply of sample containers, trip blanks, and water for field blanks, and should be informed by field personnel when supplies are low.
- 6.3.5 Soil and core sampling supplies are stored in transportainers in Building 843 area at Site 300 or may be obtained from LLNL Stores.

6.4 Operation

- 6.4.1 Samples should be collected as described in the appropriate sampling or drilling SOP.
- 6.4.2 If the samples are to be preserved, the sampler should:
 - A. Determine the approximate volume of acid (or base) needed to preserve a sample, yet still keep it safely within the criteria stated in Section 6.2.4 of this SOP.
 - B. Preserve the appropriate samples using the pre-determined amount of acid (or base).
 - C. Spot check 10% of preserved samples using pH paper or meter.
 - D. If any of the tested samples pH do not meet the preservation requirement, test all samples and add more acid (or base) as required. If any of the tested samples have a pH outside the shipping criteria (stated in Section 6.2.4 of this SOP) test all samples. If the pH is outside the criteria, the samples are considered hazardous and must be shipped by the LLNL Shipping Department as hazardous material. If the pH is within the criteria, the samples may be shipped as nonhazardous.
- 6.4.3 Samples requiring filtering (such as dissolved metals), should be performed in the field. Bailed samples should be filtered and preserved at the CAL if a filtration device is not available in the field. The CoC should describe any filtration the CAL needs to perform.
- 6.4.4 If samples that require preservation at the time of collection cannot be preserved in the field or upon returning from the field, samples are to be preserved by the CAL immediately upon receipt. This should be clearly noted on the CoC. In all cases, samples are to be preserved within 12 h from the time of collection, except in the instances where the samples can be analyzed before their unpreserved holding time.
- 6.4.5 Samples requiring refrigeration of 4°C must be protected from getting wet. Samples must be immediately placed in an ice chest containing either Blue Ice packs (in air-tight plastic bags), bagged or loose ice cubes. A temperature blank should always be included in the ice chests so the CAL can check the temperature

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of the cooler at time of sample receipt. If samples are not submitted to the CAL daily, ice chests should be checked periodically and thawed ice replaced. The ice chests should be visually inspected and if the cooler appears unclean, it should be washed with soapy water and completely rinsed with deionized water. Blue Ice plastic bags should be changed monthly to avoid potential sample contamination.

6.5 Field Post Operation

Store unused sample containers at Building 833 or Building 843 at Site 300 or return to the SC, as appropriate.

6.6 Office Post Operation

Note sample preservation methods in sampling logbook and on CoC.

7.0 QA RECORDS

7.1 Completed chain-of-custody forms

7.2 Sampling logbooks and field sheets

8.0 ATTACHMENTS

Attachment A—Recommended Sample Volumes, Containers, Holding Times, and Preservation Methods for Aqueous Samples

Attachment B—Recommended Sample Volumes, Containers, Holding Times, and Preservation Methods for Solid/Sediment Samples

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Attachment A

Recommended Sample Volumes, Containers, Holding Times, and Preservation Methods for Aqueous Samples

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Attachment A. Recommended sample volumes, containers, holding times, and preservation methods for aqueous samples.^a

Parameter	Method	Required volume and container ^b	Preservation method	Holding time
Acidity	EPA305.1	1 × 250mL, P, or G	Cool, 4°C	14 days
Anions:		1 × 500mL, P, or G	Cool, 4°C	48 hr
Bromide	EPA300.0			
Chloride	EPA300.0			
Fluoride	EPA300.0			
Nitrate as N	EPA300.0			
Nitrate as NO ₃	EPA300.0			
Nitrite as N	EPA300.0			
Nitrite as NO ₂	EPA300.0			
Phosphate	EPA300.0			
Sulfate	EPA300.0			
Biochemical oxygen demand (BOD)	EPA405.1	2 × 1L, P, or G Take sample before chlorination	Cool, 4°C	48 hr
Chemical oxygen demand (COD)	EPA410	1 × 125mL, P, or G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Chloride	EPA300.0	1 × 250mL, P, or G	None required	28 days
Chromium VI	EPA218.4, EPA218.5 EPA 7196	1 × 500mL, P, or G	Cool, 4°C	24 hr
Coliform: Fecal and total	EPA9131	1 × 250mL, P, or G	Cool, 4°C ^c	6 hr
Color	EPA110.1	1 × 125mL, P, or G	Cool, 4°C	48 hr
Conductance, specific	EPA120.1	250mL, P, or G	Cool, 4°C	28 days ^d
Cyanide, total	EPA335.2, EPA335.1	1 × 1L, P, or G	Cool, 4°C, NaOH to pH>12 ^e	14 days
Dissolved drinking water metals (except Hg): As, Ba, Cd, Cr, Pb, Se, Ag	EPA200 series	1 × 1L, P	Prefiltered, Cool, 4°C, HNO ₃ to pH<2	6 mo
Dissolved oxygen demand	EPA360.1	1 × 300mL, G bottle, and top	None required	Immediate analysis
Drinking water Volatile organics	EPA502.2, EPA524.2	3 × 40mL, G, Teflon-lined septum	Cool, 4°C	7 days
Ethylene dibromide (EDB)	EPA504	1L, AG, Teflon-lined cap	Cool, 4°C	14 days
Fish toxicity	Fathead or Stickleback	1 carboy provided by CAL	Cool, 4°C	24 hr

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Attachment A. (Continued)

Parameter	Method	Required volume and container ^b	Preservation method	Holding time
Flashpoint	–	1 × 250mL, P	Cool, 4°C	14 days
Gamma spec ^f	901.1	1L, P	HNO ₃ to pH<2	6 mo
General minerals:		2L total (see below)		
Alkalinity		1L, P	Cool, 4°C	48 hr (due to nitrates and nitrites)
Bicarbonate	EPA310.1			
Total	EPA310.1			
Carbonate	EPA310.1			
Hydroxide	EPA310.1	1L, P (metals portion)	Cool, 4°C, HNO ₃ to pH<2	6 mo
Aluminum	EPA200.7			
Calcium	EPA200.7			
Chloride	EPA300.0			
Copper	EPA200.7			
Flouride	EPA340.2			
Hardness	2320B			
Iron	EPA200.7			
Magnesium	EPA200.7			
Manganese	EPA200.7			
MBAS	EPA425.1			
Nickel	EPA200.7			
Nitrate as N	EPA345.1			
Nitrate as NO ₃	EPA345.1			
Nitrite as N	EPA353.2			
Ortho-phosphate	4500-P			
pH	EPA150.1			
Potassium	EPA200.7			
Sodium	EPA200.7			
Specific cond.	EPA120.1			
Sulfate	EPA300.0			
TDS	EPA160.1			
Zinc	EPA200.7			
Gross alpha and beta	EPA900.0	1L, P	HNO ₃ to pH<2	6 mo
HE compounds:	EPA8330	1 × 1L, AG, Teflon- lined septum	Cool, 4°C, store in dark	7 days before extraction/40 days
HMX				
RDX				
TNT				
Hydrogen ion (pH)	EPA150.1	1 × 250mL	None	12 hr
Mercury	EPA245	1L, P	HNO ₃ to pH<2	28 days
Mercury, dissolved	EPA245	1L, P	Filter, HNO ₃ to pH<2	28 days

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Attachment A. (Continued)

Parameter	Method	Required volume and container ^b	Preservation method	Holding time
Nutrients:				
Ammonia as N Total Kjeldahl N	EPA350.2 EPA351.3	500mL, P	Cool, 4°C, H ₂ SO ₄ to pH<2	Analyze as soon as possible
Nitrate as N Nitrate as NO ₃ Nitrite as N Nitrite as NO ₂	EPA300.0 EPA300.0 EPA300.0 EPA300.0	500mL, P	Cool, 4°C, unpreserved	48 hr (due to nitrates and nitrites)
NPDES metals (except Hg and CrVI): Al, Sb, As, Ba, Be, B, Cd, Cr, Co, Cu, Fe, Pb, Mn, Mo, Ni, Se, Ag, Tl, V, Zn	EPA200 series	1L, P	Cool, 4°C	6 mo
Oil and grease	EPA413.2, EPA418.1	2 × 1L, AG, Teflon- lined cap	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Orthophosphate	EPA300	125mL, P, or G	Filter immediately, Cool, 4°C	48 hr
PCBs/pesticides	EPA608, EPA615, EPA509	2 × 1L AG, Teflon- lined cap	Cool, 4°C	7 days before extraction/40 days after
Phenolics	EPA420.1, EPA9065	1L, AG, Teflon-lined cap	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Plutonium isotopes ^f		1L, P	HNO ₃ to pH<2	6 mo
Purgeable aromatics	EPA602, EPA8020	3 × 40mL, G, Teflon- lined septum	Cool, 4°C	7 days
Purgeable halocarbons	EPA601, EPA8010	3 × 40mL, G, Teflon- lined septum	Cool, 4°C	14 days
Purgeable organics	EPA624, EPA8240 EPA 8021	3 × 40mL, G, Teflon- lined septum	Cool, 4°C	7 days
Radium 226/228 ^f	904.0	2L, P	HNO ₃ to pH<2	6 mo
Semivolatile extractable organics	EPA625, EPA8270	2 × 1L, AG, Teflon- lined cap	Cool, 4°C	7 days before extraction/40 days after
Silica	EPA200.7	500mL, P	Cool, 4°C	28 days
Thorium isotopes ^f		1L, P	HNO ₃ to pH<2	6 mo

Attachment A. (Continued)

Parameter	Method	Required volume and container ^b	Preservation method	Holding time
Total dissolved solids (TDS)	EPA160.1	250mL, P, or G	Cool, 4°C	7 days
Total organic carbon (TOC)	EPA415	3 × 40mL G Teflon-lined cap	Cool, 4°C, HCl or H ₂ SO ₄ to pH<2	28 days
Total organic halides (TOX)	-	1L, G Teflon-lined cap	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Total settleable solids	EPA160.5	500mL, P, or G	Cool, 4°C	7 days
Total sulfides	EPA376.1	1L, P or G	Cool, 4°C, add 4 drops of 2N Zinc acetate per 100 mL, NaOH to pH>9	7 days
Total suspended solids (TSS)	EPA160.2	500mL, P, or G	Cool, 4°C	7 days
Total uranium ^f		250mL, P	HNO ₃ to pH<2	6 mo
TPH diesel and TBOS analysis	modified EPA8015	1L, GWM, AG	Cool, 4°C	14 days
TPH gas	modified EPA8015	3 × 40mL, G, Teflon-lined septum	Cool, 4°C	14 days
Tritium	906.0	250mL, G	Cool to 4°C	6 mo
Turbidity	EPA180.1	1 × 250mL, P, or G	Cool, 4°C	48 hr
Uranium isotopes ^f		1L, P	HNO ₃ to pH<2	6 mo

^a These volumes may vary between CALs. Each CAL should be consulted prior to use.

^b Polyethylene (P), glass (G), amber glass (AG), or glass-wide mouth (GWM).

^c Collect in sterilized bottles. If water containing residual chlorine and other halogens is to be collected, add sufficient Na₂S₂O₃ to clean sample bottle before sterilization to give a concentration of about 100mg/L in the sample. When sample is collected, leave ample air space in the bottle (at least 2.5cm).

^d If analysis is not complete within 24 hr of sample collection, sample should be filtered through a 0.45 micron filter and stored at 4°C.

^e Oxidizing agents such as chlorine decompose most cyanides. To determine whether oxidizing agents are present, test a drop of the sample with potassium iodide-starch test paper. A blue color indicates the need for treatment. Add 0.1N sodium arsenite solution a few mL at a time until a drop of sample produces no color on the indicator paper. Add an additional 5mL of sodium arsenite solution for each liter of sample. Ascorbic acid can be used as an alternative although it is not as effective as arsenite. Add a few crystals of ascorbic acid at a time until a drop of sample produces no color on the indicator paper. Then add an additional 0.6g of ascorbic acid for each liter of sample volume.

^f Sample volumes for radiological analyses may be combined in one container when the preservative is the same.

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Attachment B

Recommended Sample Volumes, Containers, Holding Times, and Preservation Methods for Solid/Sediment Samples

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Attachment B. Recommended sample volumes, containers, holding times, and preservation methods for solid/sediment samples.^a

Parameter	Method	Required volume and container ^b	Preservation method	Holding time
Alkalinity	310.1	4 oz GWM or B/ST	Cool, 4°C	14 days
Chromium VI	EPA7196, EPA7197	4 oz GWM	Cool, 4°C	24 hr
Cyanide, total	EPA9010	4 oz GWM or B/ST	Cool, 4°C	14 days
Flashpoint	EPA1010	4 oz GWM w/ TLC or B/ST	none	14 days
Gamma scan ^c	HASL 300	700g, P	None	6 mo
Gross alpha and beta ^c	9310, SM7110	25g, P	None	6 mo
HE compounds: HMX, RDX, TNT	EPA8330	8 oz GWM w/TLC or B/ST	Cool, 4°C	14 days before extraction/40 days after
Hydrongen ion (pH)	EPA9040, EPA9045	4 oz GWM or B/ST	None required	Immediate analysis
Isotopic uranium ^c	U-NAS-NS-3050	25g, P	None	6 mo
Isotopic plutonium ^c	Pu-NAS-NS-3058	25g, P	None	6 mo
Isotopic thorium ^c	Th-NAS-NS-3004	25g, P	None	6 mo
Mercury	EPA7470, EPA7471	4 oz GWM	Cool, 4°C	38 days in glass
Metals except CrVI and Hg:	EPA6000 and 7000 series	4 oz GWM	Cool, 4°C	6 mo
Nitrate	EPA9200	4 oz GWM or B/ST	Cool, 4°C	28 days
Nitrite	–	4 oz GWM or B/ST	Cool, 4°C	48 hr
Oil and grease	EPA9071	8 oz GWM w/TLC or B/ST	Cool, 4°C	28 days
Organic carbon, total (TOC)	EPA9060	20g, G or B/ST w/TLC 4 oz jar or B/ST w/TLC	Cool, 4°C	14 days
Radium 226/228 ^c	9315, HASL 300	700g, P	None	6 mo

Attachment B. (Continued)

Parameter	Method	Required volume and container ^b	Preservation method	Holding time
Semivolatile organics, Organochlorine pesticides, PCBs, and Herbicides: ^d		8 oz GWM w/TLC or B/ST	Cool, 4° C	14 days before extraction/40 days after
STLC ^e metals extraction	CAM-WET	8 oz GWM w/TLC or B/ST	Cool, 4° C	28 days
Sulfate	EPA9038	4 oz GWM or B/ST	Cool, 4° C	28 days
Sulfides	EPA9030	4 oz GWM or B/ST	Cool, 4° C, moisten sample surface w/ 2N zinc acetate	7 days
Total organic halides (TOX)	EPA9020	50g G or B/ST w/TLC	Cool, 4° C	14 days
Total uranium ^c	ASTM 5174-91, HASL 300	250g, P	None	6 mo
Tritium ^c	EERF H.01	2 × 8 oz (500 g), GWM or B/ST	None ^f	6 mo
Gasoline	EPA8015	15g G or B/ST w/TLC	Cool, 4° C	14 days
Diesel	EPA8015	300g G or B/ST w/TLC	Cool, 4° C	14 days
TPH	EPA418.1	60g	Cool, 4° C	14 days
Volatile organics ^g :		4 oz GWM w/TLC or B/ST	Cool, 4° C	14 days

^a These volumes may vary between CALs. Each CAL should be consulted prior to use.

^b Polyethylene (P), glass (G), amber glass (AG), glass-wide mouth (GWM), teflon-lined cap (TLC) or brass or steel tube (B/ST).

^c Sample volumes for Radiological analyses may be combined in one container when the preservative is the same.

^d Semi-volatile organics, organochlorine pesticides/PCBs, and herbicides may include: semivolatile organic compounds by GC/MS (EPA Method 8270), organochlorine pesticides/PCBs by GC (EPA Method 8080), total low level phenolics (EPA Method 420.1), chlorinated herbicides by GC (EPA Method 8150), herbicides by GC (EPA Method 615), TCLP semivolatile and pesticide analysis.

^e Soluble Threshold Limit Concentration extraction procedures for Hazardous Waste Identification, State of California Administrative Code, Title 22.

^f Freezing soil samples in tritium analysis is acceptable.

^g Volatile organics may include: purgeable halocarbons by GC (EPA Method 8010), purgeable aromatics by GC (EPA Method 8020), purgeable halocarbons by GC/MS (EPA Method 8240), TPH gas and diesel (modified EPA Method 8015), nonhalogenated volatile organics by GC (EPA Method 8015), fuel fingerprint by GC/MS, TCLP volatile organic analysis.